(NASA-TM-X-70381) EVALUATION OF GROUND CHARACTERIZATION SAMPLES MCN-2 AND MCN-3, SECTION 1 (NASA) 56 p HC \$6.00 CSCL 13H

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# SECTION I. EVALUATION OF GROUND CHARACTERIZATION SAMPLES MCN-2 AND MCN-3

A detailed metallurgical evaluation of two exothermically-brazed nick i ground characterization samples was conducted at Battelle-Columbus. Samples MCN-2 and MCN-3 were among six samples that were brazed as part of Exothermic Brazing Experiment M552 at the Marshall Space Flight Center in November, 1972.\* They were made to obtain base-line data for use in determining the effect of weightlessness on (1) the wetting and flow behavior of brazing filler metals and (2) the metallurgical characteristics of the brazed joints. The samples were evaluated by metallographic examination and electron microprobe analysis.

#### SAMPLE DESCRIPTION

Each sample consisted of a nickel\*\* sleeve into which a thin-wall nickel tube was brazed with the self-fluxing silver-copper filler metal Ag-28 Cu-0.2 Li (BAg-8a). The filler metal strip (0.035 inch thick by 0.145 inch wide) was formed into rings; one ring was placed in each of two grooves that were located near each end of the sleeve. The brazing clearance, i.e., the spacing between the tube and the sleeve, was 0.010 inch; the clearance was uniform for the all joint length. Spacers or inserts were used to provide this clearance and maintain the concentricity of the tube within the sleeve.

## Sectioning

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Samples MCN-2 and MCN-3 were sectioned perpendicular to the longitudinal axis of the tube assembly to produce a series of circular sections.

<sup>\*</sup> The other samples were also sent to Battelle-Columbus. After preliminary examination and sectioning, two samples were sent to the Oak Ridge National Laboratory for isotope mapping and two samples were sent to the University of Wisconsin for metallurgical study.

<sup>\*\*</sup> The composition of the bar stock from which the tube and sleeve were machined was essentially pure nickel with traces of copper and iron.

Sectioning was done with a water-cooled silicon carbide cut-off wheel, 0.020 inch wide. The use of a narrower cut-off wheel to minimize the width of the kerf was considered. However, cutting tests conducted with a 0.010-inch-wide wheel indicated that equipment modifications would be needed to produce parallel cuts and avoid wheel breakage; also, cutting rates with this wheel were very slow.

Both samples were sectioned as shown in Figure I-1. Sectioning was done in general conformance with the plan adopted during a meeting that was held at Battelle-Columbus in August, 1972. However, instead of sectioning to produce circular specimens with a uniform wiath of 0.100 inch (2.5 mm), the samples were sectioned in areas that appeared to be more informative, i.e., through the filler metal ring grooves, through porosity areas, etc. Ten sections were cut from Sample MCN-2 and nine sections were cut from Sample MCN-3. Each section was identified to indicate its location along the longitudinal axis of the sample.

#### Visual Examination

The sections from Samples MCN-2 and MCN-3 were examined under low-power magnification to (1) study the visual appearance of the brazed tube assembly, (2) confirm the radiographic findings, and (3) select sections for metallographic examination. The results of these studies are discussed below.

# Sample MCN-2

As indicated by radiography, this tube assembly was well brazed from one end to the other. Filler metal was present around the entire joint circumference in each section (except those that were cut through the filler metal ring grooves). The joint quality was excellent, and there were no visible defects in any of the sections. Excess filler metal was accumulated at the bottom of each ring groove; i.e., at the 6 o'clock position of the sample during brazing. However, excellent flow was indicated by the presence of a thin layer of filler metal over the entire surface of the ring grooves. Five sections were selected for metallographic study.

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To aid in studying the flow characteristics of the filler metal, a short section of one filler metal ring in each sample was removed, irradiated with neutrons to produce radioisotope  $Ag^{110}$ , and replaced in the groove. The isotope was located in register with axial lines that were machined on the outer surface of the sleeve to fix the isotope position with respect to the sample orientation during brazing. The isotope activity level of Samples MCN-2 and MCN-3 was slightly less than 50 microwires.

Samples MCN-2 and MCN-3 were almost identical. Both samples were brazed in the horizontal position; however, the samples were oriented during brazing to locate the isotope tracers in MCN-2 and MCN-3 at 3 o'clock and 6 o'clock, respectively. The samples were brazed in a vacuum furnace with exotherms that weighed 60 grams; the pressure within the furnace was 8.5 x 10<sup>-5</sup> torr for MCN-2 and 9.8 x 10<sup>-5</sup> torr for MCN-3. The brazing temperature was monitored with a thermocouple attached to the center ID of the tube. The maximum temperature to which the samples were heated during brazing was 1930 F (1054.4 C) for MCN-2 and 1915 F (1046.1 C) for MCN-3.

After brazing, the samples were radiographed at the Marshall Space Flight Center. The following comments are based on the interpretation of the radiographs.

Sample MCN-2: Excess filler metal puddled in the bottom, i.e., 6 o'clock position, of the ring grooves — excellent joint quality — no porosity.

Sample MCN-3: Excess filler metal puddled in the bottom of one ring groove -- some porosity in one location.

### VISUAL EXAMINATION

The sections from Samples MCN-2 and MCN-3 were examined under low-power magnification to (1) study the visual appearance of the brazed tube assembly, (2) confirm the radiographic findings, and (3) select sections for metallographic examination. The results of these studies are discussed below.

#### Sample MCN-2

As indicated by radiography, this tube assembly was well brazed from one end to the other. Filler metal was present around the entire joint

circumference in each section (except those that were cut through the filler metal ring grooves). The joint quality was excellent, and there were no visible defects in any of the sections. Excess filler metal was accumulated at the bottom of each ring groove; i.e., at the 6 o'clock position of the sample during brazing. However, excellent flow was indicated by the presence of a thin layer of filler metal over the entire surface of the ring grooves. Five sections were selected for more detailed metallographic study.

## Sample MCN-3

This tube assembly was also well brazed for its entire length, and the circum/erential joints in all sections (excluding sections cut through the ring grooves) were filled with brazing filler metal. While the joint quality was generally excellent, there was evidence of porosity or voids in the joint contained in one section. The porous area was located to the left of the right-hand ring groove in the 1 or 2 o'clock position during brazing. The existence of porosity in this location was also indicated by radiography. Excess filler metal was accumulated at the bottom of each ring groove; there was considerably more filler metal in the right-hand groove than in the lett-hand groove. The surfaces of each ring groove were coated with a thin layer of filler metal. Five of these sections were also selected for further study.

#### METALLOGRAPHIC EXAMINATION

Sections from Sample MCN-2 and MCN-3 were examined metallographically to study (1) the wetting and flow characteristics of the filler metal, (2) the quality of brazing, (3) defects and their frequency of occurrence, (4) metallurgical reactions between the base metal and filler metal, and (5) joint microstructures. These studies are discussed below.

## Sample MCN-2

Five sections representing areas where differences in the joint Characteristics might be evident were examined. The designation and approximate location of each section are shown in Figure I-2. Sections MCN-2-1

and MCN-2-9 were cut and mounted to permit examination of the joint surfaces parallel to the longitudinal axis of the sample. The remaining sections were mounted to show the entire circumferential joint between the tube and sleeve, i.e., the surface perpendicular to the longitudinal axis of the sample. After polishing, the metallographic specimens were examined in the unetched and etched condition; the metallurgical features of the joints were generally most evident in the unetched condition.

## Section MCN-2-1

One side of the joint in this section is shown at a magnification of 20% in Figure I-3. The joint quality is excellent; the filler metal wet the base metal well and flowed to completely fill the joint between the tube and sleeve. The insert used to maintain the spacing between the tube and sleeve was brazed to the remainder of the assembly. A few isolated voids were present in the joint area. The other side of the joint had an appearance similar to that shown in Figure I-3.

Selected areas of this joint are shown in the as-polished condition at 250X and 500X in Figures I-4 and I-5, respectively; the area covered by Figures I-4 is also shown in the etched condition at the same magnification in Figure I-6. The microstructure between the joint interfaces is largely comprised of a eutectic phase and a phase that appears to have been formed by the reaction of the filler metal with the base metal. Etching appeared to have the same effect on this phase as it did on the phase along the joint interface. The dissolution of the base metal by the molten filler metal is clearly evident in the attached photomicrographs. The width of the zone resulting from this reaction was about 0.001 inch.

The microstructure of the brazed joints is discussed in more detail later in this report.

## Section MCN-2-2

The full circumferential joint between the nickel tube and sleeve is shown at 4X in Figure I-7. The quality of this joint was generally excellent except for an accumulation of voids that are evident at the 12

o'clock and 3 o'clock locations in Figure I-7. (The 3 o'clock position corresponds to the location of the isotope tracer in this sample.) These voids, one of which extended across 70 percent of the joint, could have formed as the result of insufficient filler metal in this area. They could have also formed if filler metal drained from this area into an area deficient in filler metal. Their presence was not noted in the interpretation of the radiographs of this sample. Voids at the 3 o'clock location are shown at 250% in Figure I-8.

Interface-to-interface measurements were made at 90 degree intervals around the joint circumference to determine (1) the clearance between the tube and sleeve and (2) the concentricity of the tube within the sleeve. These measurements are listed below.

Location	Clearance, in.
12 o'clock	0.0093
3 o'clock	0.0078
6 o'clock	0.0099
9 o'clock	0.0109

The average joint clearance, 0.0095 inch, was just slightly less than the design clearance of 0.010 inch. The tube was well-centered vertically in the sleeve, but was offset slightly (toward the 3 o'clock location) in the horizontal orientation.

#### Section MCN-2-3

A section of the right-hand filler metal ring groove is shown at 4X in Figure I-9. The accumulation of filler metal at the bottom of the ring groove indicates that the tube assembly was brazed in the orientation shown in this figure. Capillary forces were ineffective because the ring groove was too wide (0.055 inch). As a result, the filler metal collected in a puddle due to gravity and it did not form a smooth meniscus. Nevertheless, sufficient flow occurred to coat the entire surface of the ring groove with a thin film of filler metal.

A section near the left-hand edge of the accumulated filler metal is shown at 100% in Figure I-10. There are some isolated voids in this area. The microstructure is largely comprised of a eutectic phase and another phase resulting from the reaction of the filler metal with the base metal.

# Section MCN-2-6

The entire circumferential joint in a section midway between the ring grooves was examined; its appearance is similar to that of the joint shown in Figure I-7. The joint quality was generally excellent; the filler metal wet the base metal and filled the joint almost completely. There were a few isolated voids in the joint; they increased in size and number in the 3 to 6 o'clock quadrant. The joint microstructure was similar to that of the section discussed above.

## Section MCN-2-9

A complete cross section of the left-hand ring groove is shown at 20% in Figures I-lla and I-llb. In the section of the joint that was located at the 12 o'clock position during brazing (Figure I-lla), there was a fillet of filler metal in the corners of the ring groove, but the groove itself was empty. However, the surfaces of the groove were coated with a thin film of filler metal as it flowed into the joint between the tube and sleeve. In the section that was located at 6 o'clock during brazing (Figure I-llb), the ring groove was completely filled with accumulated filler metal as indicated by the radiograph of this area.

#### Sample MCN-3

Five representative sections from Sample MCN-3 were examined metallographically. The designation and approximate location of each section are shown in Figure I-12. Three sections (MCN-3-3, MCN-3-5, and MCN-3-9) were cut and mounted to permit examination of the joint surfaces parallel to the longitudinal axis of the sample. The remaining sections were mounted to show the full circumferential joint between the tube and sleeve.

#### Section MCN-3-2

A circumferential section through the right-hand ring groove of this sample is shown at 4X in Figure I-13. The location of the accumulated filler metal indicates that the tube assembly was brazed in the orientation shown in Figure I-13. Considerably more filler metal remained in the ring groove of this sample after brazing than in the corresponding ring groove of Sample MCN-2 (Figure I-9). Apparently, the filler metal did not flow as well as it did in the previous sample. As observed previously; however, the surfaces of the ring groove were coated with a thin film of filler metal.

The microstructure of the accumulated filler metal is shown at 100% in Figure I-14. It is similar in appearance to microstructures examined previously.

## Section MCN-3-3

The cross section of the right-hand ring groove was also examined. In appearance, this cross section was essentially identical to that shown in Figures I-lla and I-llb. The lower portion of the ring groove was filled with filler metal; the upper portion was empty. However, filler metal had flowed from the ring groove to fill the joint between the tube and sleeve.

## Section MCN-3-4

A circumferential section between the ring grooves is shown at 4X in Figure I-15. The joint between the tube and the sleeve is essentially void for about 40 degrees of its circumference, and there are numerous voids on either side of this area. The void area was located at the top of the sample during the brazing cycle. In the radiographs of this joint, it was interpreted as an area of porosity. The remainder of this joint was largely defect-free.

A section of the void area is shown at 250X in Figure I-16. The presence of some filler metal and some material in a zone where the molten filler reacted with the base metal implies that filler metal was present in this area during the brazing cycle. However, much of it (presumably the eutectic phase) drained into areas deficient in filler metal before solidification was completed. There is one area along the lower interface where the filler metal appears to have penetrated the boundaries of a grain in the base metal.

A defect-free area of this joint is shown at 250X and 500X in Figure I-17. The joint microstructure is similar to that observed in various sections of Sample MCN-2. However, the length of the particles along the interface indicates that more reaction between the molten filler metal and the base metal occurred in this sample than in Sample MCN-2.

## Section MCN-3-5

The section adjacent to MCN-3-4 was examined in cross section. The continuation of the area that is essentially void can be seen in the upper section of this joint (Figure I-18a). This area has the appearance of being once filled with filler metal during the brazing cycle. The lower section of this joint, i.e., the section located at 6 o'clock during brazing, is completely filled with filler metal (Figure I-18b). Numerous isolated voids are present in the joint.

#### Section MCN-3-9

This section was mounted to permit examination of the cross section of the left-hand ring groove. The ring groove in the area located at 12 o'clock during brazing was empty; the bottom of the ring groove was partially filled with filler metal. The joint microstructure was similar to that observed in other sections of this sample.

#### MICROSTRUCTURAL STUDIES

An investigation was conducted to study the base metal-filler metal reactions that occurred during brazing and their effects on the microstructure of the brazed joint. Also, the approximate composition of some of the phase constituents was determined.

The microstructure of a typical area along the joint interface of Section MCN-2-1 is shown in the unetched condition at a magnification of 250X in Figure I-19. Considerable reaction between the base metal and the molten filler metal occurred during the brazing cycle. In Section MCN-2-1, measurements indicated that a layer of base metal, 7.0005-inch-thick or less, was

dissolved by the molten filler metal to form another phase at the joint interface (areas labelled A in Figure I-19). Presumably, this is a nickel-copper phase because nickel and copper form a continuous series of solid solutions; on the other hand, the solubility of nickel in liquid silver is less than 0.5 weight percent at the brazing temperature. (1,2) Based on its appearance in the unetched and etched condition, this phase also appears to be present in the filler metal matrix (areas labelled B in Figure I-19).

The microstructure of the brazed joint was further studied by electron microprobe analysis. An area\* similar to that indicated in Figure I-19 was scanned with the electron beam to determine the distribution of the following elements: nickel, copper, and silver. The resulting X-ray images for these elements are shown in Figures I-20a, I-20b, and I-20c. By examining the variations in intensity that indicate the approximate concentration distribution of each element in the area being scanned, it was possible to estimate the composition of phase constituents in areas of interest. The results of electron microprobe analysis are discussed below.

- (1) The approximate composition of the phase (A in Figure I-19) at the joint interface that resulted from dissolution of the base metal by the molten filler metal is 95 Cu-5 Ni.
- (2) The composition of the phase (B in Figure I-19) in the filler metal matrix is identical to that of the phase at the joint interface. This copper-rich reaction product formed along the interface and became detached as dissolution progressed.
- (3) The light areas of the filler metal matrix (C in Figure I-19) are composed of essentially pure silver. These areas are devoid of copper because of the reaction of the molten filler metal with the base metal.
- (4) Attempts were made to identify other constituents of the microstructure, e.g., areas labelled D. However, the composition of these areas could not be determined because they were too small. Presumably, they represent a copper-rich phase similar to areas A and B.

Hansen, M., Constitution of Binary Alloys, McGraw-Hill Book Company, New York, N.Y. (1958).

Elliot, R. P., Ed., Constitution of Binary Alloys, First Supplement, McGraw-Hill Book Company, New York, N.Y. (1965).

<sup>\*</sup> The area scanned by the electron beam differed slightly from that shown in Figure I-19 because the specimen was repolished before examination. However, the area scanned contained the features shown in Figure I-19.

The exothermic brazing cycle differs substantially from that used for conventional brazing operations with Ag-28 Cu-0.2 Li filler metal. Ground characterization samples MCN-2 and MCN-3 were brazed at temperatures above 1900 F (1037.8 C); that is, at temperatures much higher than the liquidus temperature of Ag-28 Cu-0.2 Li filler metal, 1400 F (760.0 C). The time required to heat these samples to the brazing temperature was very short (one minute) and the total time during which the filler metal was molten was correspondingly short also. In contrast, most assemblies made with this iller metal are brazed at 1450 to 1500 F (787.7 to 815.5 C). While the time required to braze the assembly depends on its mass and the capabilities of the furance, it is relatively long in comparison with the time required for exothermic brazing. Since the amount of reaction between the base metal and filler metal is largely dependent on the brazing temperature and the time during which the filler metal is molten, the microstructures of exothermicallybrazed joint could differ appreciably from those of joints brazed conventionally. To study this possibility, a nickel-tee joint was brazed with Ag-28 Cu-0.2 Li filler metal in a vacuum (2.8 x 10<sup>-5</sup> torr) at 1500 F for 5 minutes.\*

An unetched section of the nickel tee-joint is shown at 250% in Figure I-2la; for direct comparison, a section from MCN-2 is shown at the same magnification in Figure I-2lb (this figure is identical to I-4). The microstructures of these joints reflect differences attributable to the brazing conditions. The amount and distribution of the copper-rich phase (A in Figure I-2la and I-2lb) indicates that more reaction between the base metal and filler metal occurred in the exothermically brazed joint than in the furnace brazed joint. However, the width of the reaction zone at the interface was comparable for both joints. The microstructure of areas at some distance from the joint interface differs considerably. In the furnace-brazed joint (Figure I-2la), there is a silver-rich dendritic phase (C) dispersed in a matrix of the silver-copper extectic (D). In the exothermic-brazed joint (Figure I-2lb), the following phases are present: a copper-rich phase (A), a phase that is essentially pure silver (B), and a phase that was not identified (C).

The differences in the microstrucutres of exothermically-brazed and furnace-brazed joints appear to be primarily attributable to the difference in the brazing thermal cycle.

<sup>\*</sup> The material for the tea-joint was obtained from excess stock on Sample MCN-2; the filler metal was obtained from MSFC.

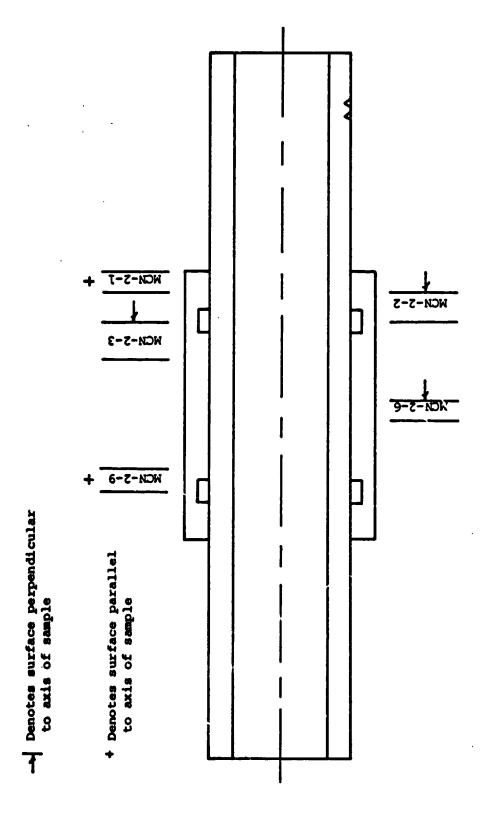


FIGURE 1-2. SURPACES EXAMINED DURING METALLOGRAPHIC EVALUATION (MCN-2)

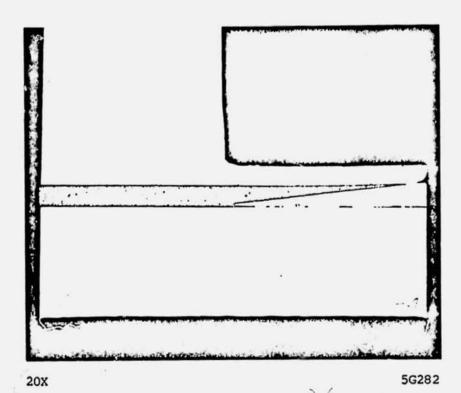


FIGURE 1-3. SECTION MCN-2-1

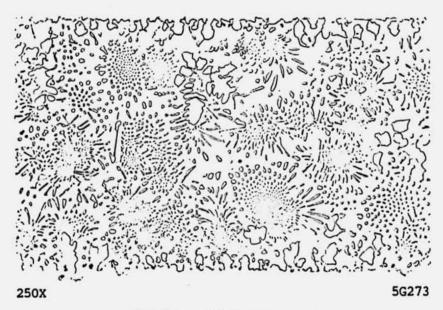


FIGURE 1-4. SECTION MCN-2-1

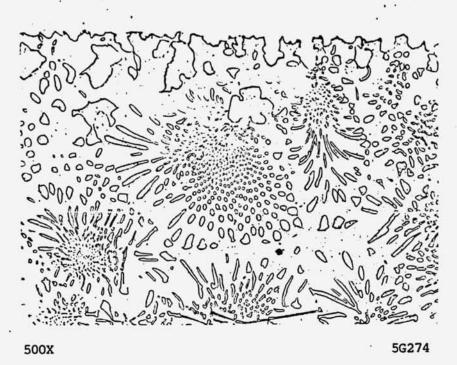


FIGURE 1-5. SECTION MCN-2-1

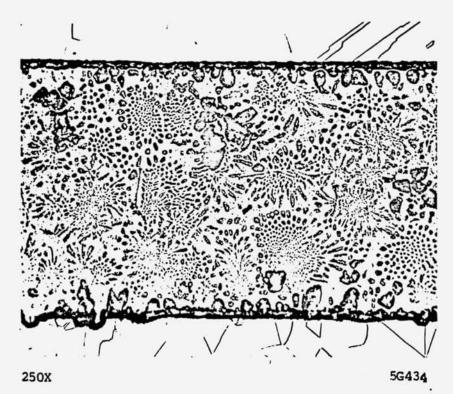


FIGURE I-6. SECTION MCN-2-1

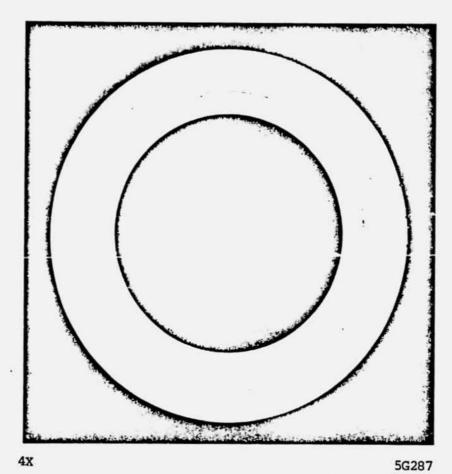
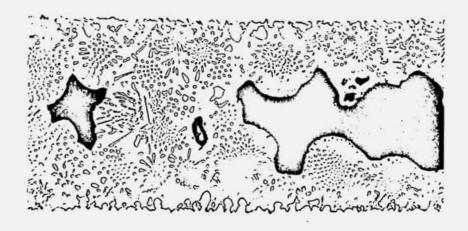


FIGURE 1-7. SECTION MCN-2-23 mm

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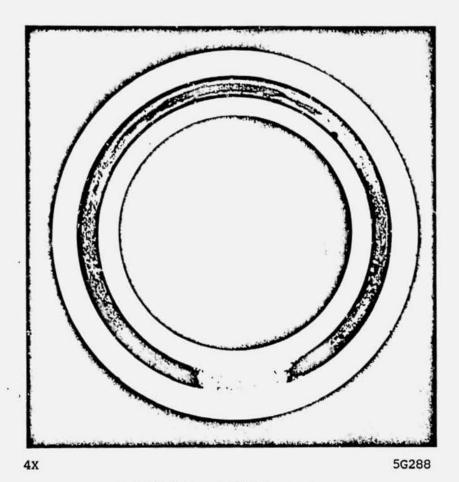
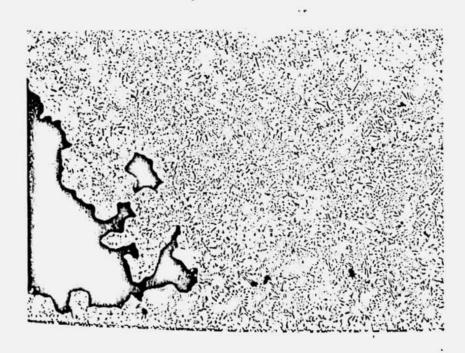
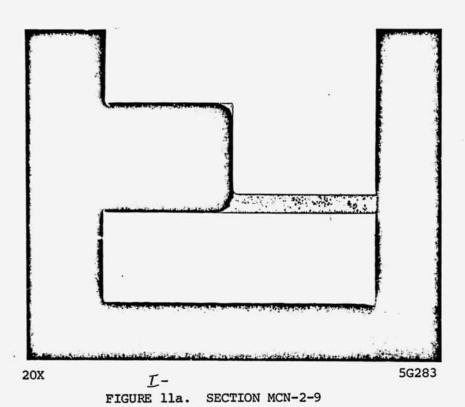


FIGURE I-9. SECTION MCN-2-3



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FIGURE I-10. SECTION MCN-2-3



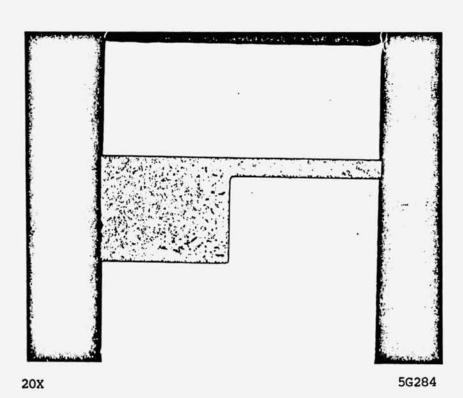
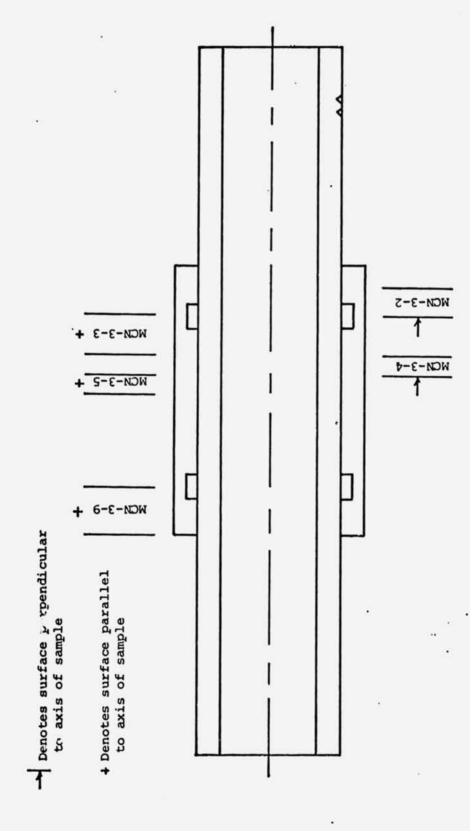


FIGURE I-11b. SECTION MCN-2-9

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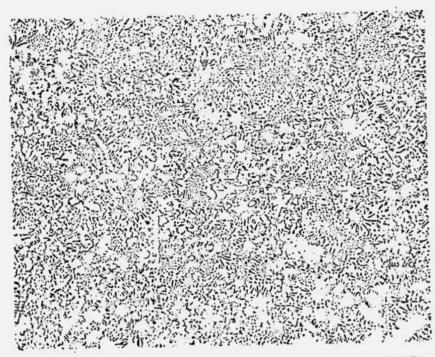


SURFACES EXAMINED DURING METALLOGRAPHIC EVALUATION (MCN-3) FIGURE 1-12.

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FIGURE I-13. SECTION MCN-3-2



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FIGURE I-14. SECTION MCN-3-2

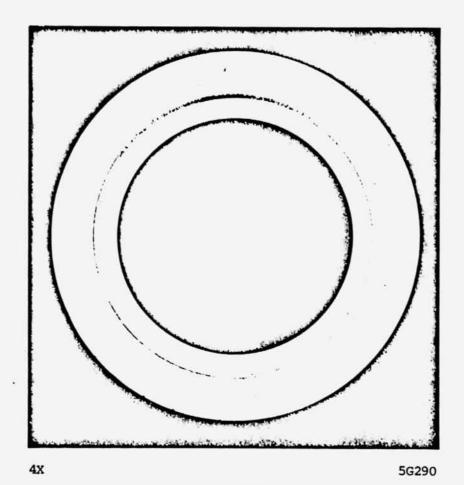
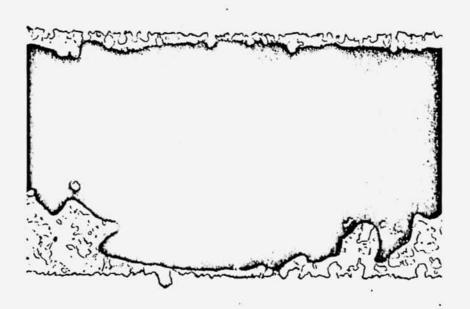


FIGURE I-15. SECTION MCN-3-4



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FIGURE I-16. SECTION MCN-3-4

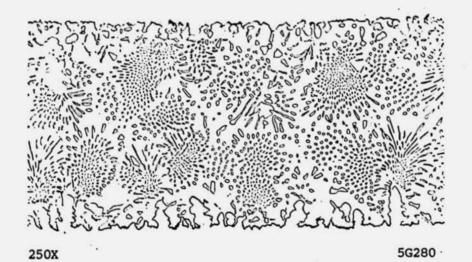


FIGURE I-17a. SECTION MCN-3-4

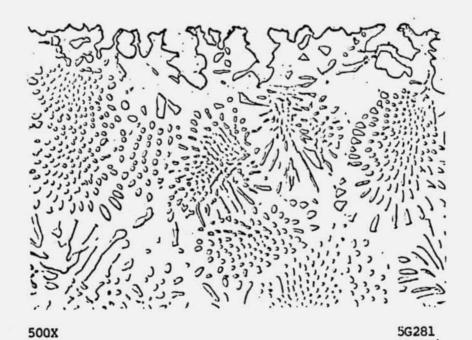


FIGURE 1-17b. SECTION MCN-3-4



FIGURE 18a. SECTION MCN-3-5

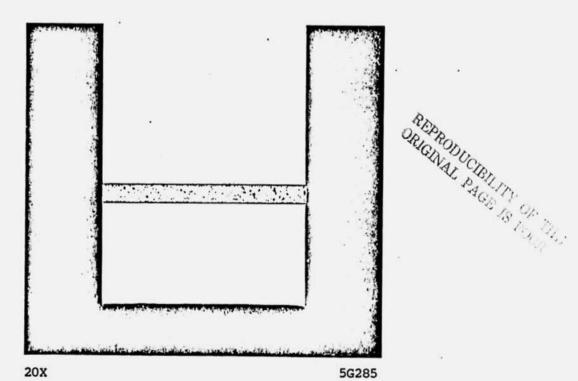


FIGURE 18b. SECTION MCN--3-5

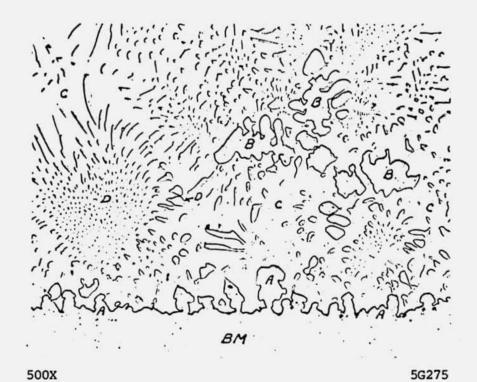


FIGURE I-19. SECTION MCN-2-1

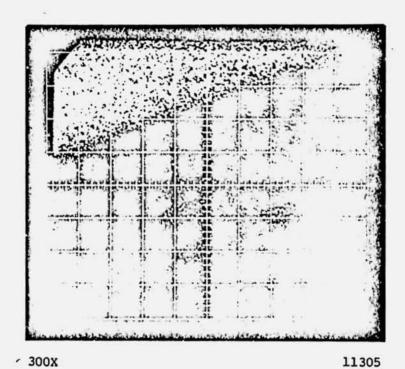


FIGURE I-20a. NICKEL X-RAY IMAGE

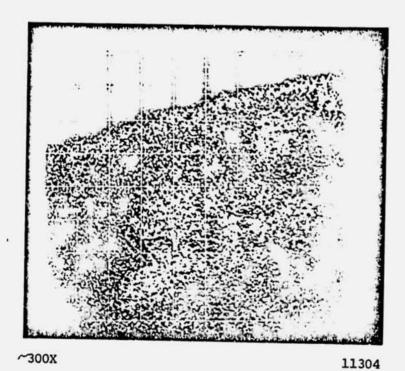


FIGURE I-20b. COPPER X-RAY IMAGE

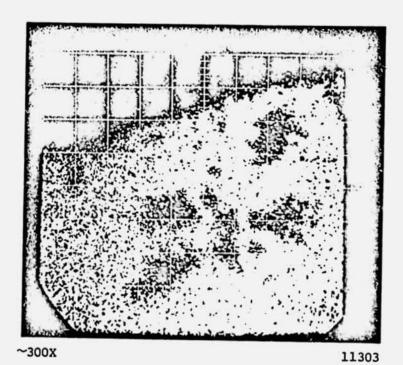


FIGURE I-20c. SILVER X-RAY IMAGE

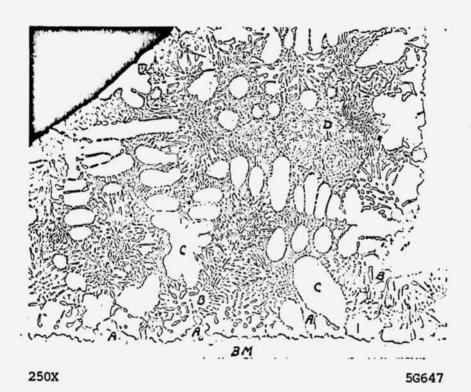
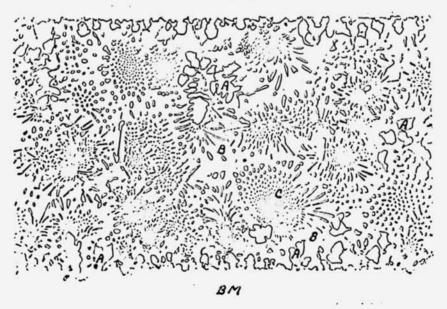


FIGURE 21a. TEE JOINT

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FIGURE 1-21b. SECTION MCN-2-1

# SECTION II. EVALUATION OF GROUND CHARACTERIZATION SAMPLES MCS-1, MCS-2, and MCS-3

A detailed metallurgical evaluation of three exothermically-brazed, stainless steel ground characterization samples was conducted at Battelle-Columbus. Samples MCS-1, MCS-2, and MCS-3 were among semanples that were brazed as part of Exothermic Brazing Experiment M552 at the Marshall Space Flight Center in January, 1973.\* They were made to obtain pase-line data for use in determining the effect of weightlessness on (1) the wetting and flow behavior of brazing filler metals and (2) the metallurgical characteristics of the brazed joints. The samples were evaluated by metallographic examination and electron microprobe analysis.

#### SAMPLE DESCRIPTION

Each sample consisted of a 304L S.S.\*\* sleeve into which a thin-wall 304L S.S. tube was brazed with the self-fluxing silver-copper filler metal Ag-28 Cu-0.2 Li (BAg-8a). The filler metal strip (0.035 inch thick by 0.145 inch wide) was formed into rings; one ring was placed in each of two grooves that were located near each end of the sleeve. The brazing clearance, i.e., the spacing between the tube and the sleeve, was 0.005 inch; the clearance was uniform for the full joint length. Spacers or inserts were used to provide this clearance and maintain the concentricity of the tube within the sleeve. The tube was slit at its center.

<sup>\*</sup> The other samples were also sent to Battelle-Columbus. After preliminary examination and sectioning, three samples were sent to the University of Wisconsin for metallurgical study.

<sup>\*\*</sup> The composition of the bar stock from which the tube and sleeve were machined was 11.46 Ni-18.70 Cr-1.8 Mn-0.44 Si-0.014 S-0.030 P-0.036 C-Fe (rest).

Samples MCS-1 and MCS-2 were almost identical. Both samples were brazed in the horizontal position in a vacuum furnace with exotherms that weighed 60 grams. The sample temperature was monitored by thermocouples attached in the following locations: (1) inside the tube at the center of the sample, (2) inside the tube at the end of the sleeve, and (3) outside and at the end of the tube. The pressure within the vacuum furnace and maximum temperature (measured at the center ID of the tube) are indicated below.

MCS-1: 
$$3.5 \times 10^{-5}$$
 torr and 1915 F (1046.1 C)  
MCS-2:  $2.6 \times 10^{-5}$  torr and 1910 F (1043.3 C)

Sample MCS-3 was brazed under the same conditions as Samp'es MCS-1 and MCS-2. However, as indicated below, the pressure and temperature at which brazing occurred varied from the planned schedule because of an equipment malfunction.

MCS-3: 
$$1.5 \times 10^{-4}$$
 torr and 1810 F (987.7 C)

After brazing, the samples were radiographed at the Marshall Space Flight Center. The following comments are based on the interpretation of the radiographs.

<u>Sample MCS-1</u>: Voids at center and both ends of sleeve; excess filler metal in bottom tube; no filler metal in ring grooves.

<u>Sample MCS-2</u>: Voids at center and both ends of sleeve; excess filler metal in bottom of tube; no filler metal in ring grooves.

<u>Sample MCS-3</u>: Voids at both ends of sleeve; excess filler metal in bottom of tube; no filler metal in ring grooves.

## SECTIONING

Samples MCS-1, MCS-2, and MCS-3 were sectioned perpendicular to the longitudinal axis of the tube assembly to produce a series of circular sections. Sectioning was done with a water-cooled silicon carbide cut-off wheel, 0.020 inch wide. The use of a narrower cut-off wheel to minimize

the width of the kerf was considered. However, cutting tests conducted with a 0.010-inch-wide wheel indicated that equipment modifications would be needed to produce parallel cuts and avoid wheel breakage; also, cutting rates with this wheel were very slow.

Both samples were sectioned as shown in Figure II-1. Sectioning was done in general conformance with the plan adopted during a meeting that was held at Battelle-Columbus in August, 1972. However, instead of sectioning to produce circular specimens with a uniform width of 0.100 inch (2.5 mm), the samples were sectioned in areas that appeared to be more informative, i.e., through the filler metal ring grooves, through porosity areas, etc. Nine: ctions were cut from each sample. Each section was identified to indicate its location along the longitudinal axis of the sample.

#### VISUAL EXAMINATION

The sections from Samples MCS-1, MCS-2, and MCS-3 were examined under low-power magnification to (1) study the visual appearance of the brazed tube assembly, (2) confirm the radiographic findings, and (3) select sections for metallographic examination. The results of these studies are discussed below.

#### Sample MCS-1

This tube-sleeve assembly appeared to be well brazed from one end to the other. Filler metal was present around the entire joint circumference in each section (except sections that were cut through the filler metal ring grooves). The joint quality was visually excellent; there was one void near the center of the sample at the 12 o'clock position during brazing. Excess filler metal was accumulated inside the tube at the 6 o'clock position. The ring grooves were empty; however, excellent flow was indicated by the presence of a very thin film of filler metal over the ring groove surface. One thermocouple was brazed on the inside of the tube. Four sections were selected for metallographic study.

## Sample MCS-2

This tube-sleeve assembly was also well brazed for its entire length, and the circumferential joints in all sections (excluding sections cut through the ring grooves) were filled with filler metal. The joint quality was generally excellent; however, there were some voids between the tube and sleeve in the far right-hand section, i.e., the section nearest the end of the tube with the annular machined grooves. These voids were also visible on the next section. Both ring grooves were empty, but they were coated with a thin film of filler metal. Filler metal was accumulated at the bottom of the tube. Some filler metal was also present in the center slit in the tube. Three sections were selected for metallographic study.

## Sample MCS-3

This assembly was well brazed for its entire length, and the circumferential joints in all sections (excluding those cut through the ring grooves) were filled with brazing filler metal. The joint quality was generally good; there were some voids between the tube and sleeve in a section near the left-hand end of the sleeve. Both ring grooves were essentially empty; there appeared to be a very small fillet in the ring grooves and the surfaces of both were coated with a film of filler metal. Filler metal was accumulated in the bottom of the tube. The surface of the accumulated filler metal was extremely rough and there were numerous areas of porosity. The surface of the accumulated filler metal in Samples MCS-1 and MCS-2 was smooth and there was no evidence of porosity. Four samples were selected for metallographic examination.

#### METALLOGRAPHIC EXAMINATION

Sections from Samples MCS-1, MCS-2, and MCS-3 were examined metallographically to study (1) the wetting and flow characteristics of the filler metal, (2) the quality of brazing, (3) defects and their frequency of occurrence, (4) metallurgical reactions between the base metal and filler metal, and (5) joint microstructures. These studies are discussed below.

# Sample MCS-1

Four sections representing areas where significant joint features might be evident were examined. The designation and approximate location of each section are shown in Figure II-2. Sections MCS-1-1 and MCS-1-2 were cut and mounted to permit examination of the joint surfaces parallel to the longitudinal axis of the sample. Sections MCS-1-4 and MCS-1-8 were mounted to show the entire circumferential joint between the tube and the sleeve, i.e., the surface perpendicular to the longitudinal axis of the sample. The metallographic specimens were examined in the unetched and etched condition.

#### Section MCS-1-1

The upper and lower sides of this joint are shown at 20X in Figures II-3a and II-3b, respectively. The upper side of this joint is completely filled and the joint quality is excellent. Excellent flow of the filler metal is also indicated in the bottom section of this joint; however, as indicated by radiography, the joint was partially void. The insert that is used to insure the concentricity of the tube within the sleeve is well-brazed also.

Selected areas of this joint are shown in the unetched condition at magnifications of 250X and 500X in Figures II-4a and II-4b. The microstructure between the joint interfaces appears to consist of a three-phase structure. The dissolution of the base metal by the molten filler metal is clearly evident. The width of the joint between interfaces is 0.0044 inch; some products of the base metal-filler metal reaction extend about 0.001 inch from the interface.

The joint microstructure and the phases contained therein are discussed in more detail later in this section.

#### Section MCS-1-2

The upper and lower portions of the section adjacent to the one discussed above (MCS-1-1) are shown at 20X in Figures II-5a and II-5b; this

section contains the filler metal ring groove and a short length of tube-tosleeve joint. The ring groove is devoid of filler metal; however, there was
evidence of a very thin film of filler metal on the ring groove surfaces
at ...gh magnification. As in the previous section (and as indicated by
radiography), the upper section of the joint was filled with filler metal;
the lower section was void. Filler metal is puddled at the bottom inside
of the tube. This filler flowed from the ring groove to the center of the
sample, through the slits in the tube, and back along the inside of the tube.

The microstructure of an area of accumulated filler metal is shown in the unetched condition at 250X and 500X in Figures II-6a and II-6b. Reaction between the base metal and molten filler metal at the interface is clearly evident. The microstructure of the bulk filler metal is quite complex, consisting of products from the base metal-filler metal reaction and possibly some silver-copper eutectic.

## Section MCS-1-4

The full circumferential joint between the tube and the 'eeve is shown at 4X in Figure 7. The joint quality is generally excellent except for two void areas in the upper right-hand quadrant. Filter metal has accumulated at the bottom inside of the tube (the macrogramme lightly out of the orientation in which brazing occurred) and it has flowed to envelop the thermocouple.

A section of void area is shown at 100X in Figure II-8. The entire area between the joint interface appears to have the once filled with filler metal; the void occurred when filler metal draines. Them this area into others that were deficient in filler metal.

An area of the filler metal accumulated inside the tube is shown at 250X in Figure II-9. The microstructure of this area is somewhat different than that shown in Figure II-6a; a dendritic phase is present in a matrix of filler metal. The amount of reaction between the base metal and molten filler metal is comparable in both areas.

One of the thermocouple wires is shown at 250X in Figure II-10. As indicated, filler metal flowed around the inside of the tube to encase these wires. Considerable reaction between the molten filler metal and the thermocouple wire occurred; the maximum depth of reaction is about 0.0008 inch.

#### Section MCS-1-9

A section through the left-hand filler metal ring groove is slown at 4X in Figure II-11. The ring groove is empty; however, its surfaces are covered with a very thin film of filler metal. Finler metal has accumulated at the bottom inside of the tube. The microstructure of the accumulated filler metal is similar to that observed in other sections of this joint.

## Sample MCS-2

Three representative sections from this sample were examined; their location and designation are indicated in Figure II-12. Two sections (MCS-2-4 and CCN-2-7) were cut and mounted to permit examination of the entire circumferential joint between the tube and sleeve, i.e., the surface perpendicular to the longitudinal axis of the sample. The other section (MCS-2-9) was cut and mounted so that the joint surfaces parallel to the longitudinal axis of the sample could be examined.

#### Section MCS-2-4

The full circumferential joint between the tube and sleeve is shown at 4X in Figure II-13. The joint quality was excellent and there were no void areas of significance. A section of this joint is shown in the unetched condition at 250X in Figure II-14a; the same section is shown etched in Figure II-14b. The joint microstructure appears similar to those observed in sections from Sample MCS-1.

Filler metal has accumulated on the inside bottom of the tube. The microstructure of an area of accumulated filler metal is shown in Figure II-15 at 250X. There appears to be little reaction at the interface between the filler metal and base metal; the microstructure is comprised primarily of the silver-copper eutectic.

## Section MCS-2-7

The circumferential joint between the tube and sleeve is shown at 4X in Figure II-16. Excellent flow of the filler metal during brazing occurred. The joint is completely filled; there were a few small isolated voids of little significance. Filler metal flowed through the slits in the center of the tube and accumulated in the bottom inside of the tube. As it did so, it brazed the thermocouple to the inside of the tube. The joint microstructure and the microstructure of the accumulated filler metal is similar to those observed previously.

#### Section MCS-2-9

The lower half of the joint in this section is shown at 4X in Figure II-17. Excellent flow of the filler metal occurred during brazing; the joint between the tube and sleeve is completely filled and the insert is brazed into the remainder of the assembly. The filler metal ring groove is essentially empty. A few isolated voids are visible in the joint. The appearance of the upper section of this joint is similar to that shown in Figure II-17.

# Sample MCS-3

Four sections representing typical joint areas in this tube assembly were examined metallographically; the location and designation of each section are shown in Figure II-18a. Three sections were cut and mounted to permit examination of the joint surfaces parallel to the longitudinal axis of the sample. The remaining section was mounted to permit examination of the entire circumferential joint.

#### Section MCS-3-2

The lower half of this section is shown at 20% in Figure II-18b. The ring groove is essentially void except for some filler metal remaining in one corner. This section of joint between the tube and sleeve is completely void; however, the joint in the upper half of this section was well filled with filler metal. The presence of voids in this area was indicated by radiography. As observed previously with Samples MCS-1 and MCS-2, filler metal accumulated on the bottom inside of the tube during brazing. However, the surface of the accumulated filler metal was extremely rough and there were numerous indications of voids or porosity. In contrast, the surface of the accumulated filler metal in the other two samples (MCS-1 and MCS-2) was very smooth. Due to an equipment malfunction, the maximum temperature to which Sample MCS-3 was heated during brazing was about 100 F (37.8 C) lower than the temperature to which the other samples were heated. Apparently, the surface roughness is associated with the manner in which the filler metal flowed and solidified.

## Section MCS-3-3

A section through the right-hand ring groove is shown at 4X in Figure II-19a. The ring groove is void; however, a thin film of filler metal was present on its surfaces. The surface roughness of the filler metal that accumulated inside the tube during brazing is clearly evident. An area of accumulated filler metal is shown at 100X in Figure II-19b. Voids or pores are present in this area. The microstructure of this area is somewhat different from that observed in similar sections from Samples MCS-1 and MCS-2. Although the maximum brazing temperature for Sample MCS-3 was less than it was for the other two samples, the products of the reaction between the base metal and the molten filler metal appear to be dispersed in larger amounts throughout the microstructure.

## Section MCS-3-5

The upper and lower portions of the section adjacent to the slits in the center of the tube are shown at 20% in Figures II-20a and II-20b. In Figure II-20a, there is a more or less continuous void in the joint between

the tube and sleeve. However, it appears that filler metal was present in this area, and that it drained away during the solidification process. The bottom joint, i.e., the area at the 6 o'clock position during brazing, was completely filled with filler metal (Figure II-20b). Fillor metal has also accumulated inside the tube and along the wall of the slit; the slit is not visible in Figure II-20a because it is not continuous around the circumference of the tube.

#### Section MCS-3-9

The upper and lower sections of this joint are shown at 20% in Figures II-2la and II-2lb; this section is located at the far left-hand side of the sample and it includes a portion of the filler metal ring groove. The joint between the tube and the sleeve is well-brazed; there were a few isolated voids of no significance. The ring groove is essentially void. The surface roughness of the filler metal accumulated inside the tube is evident in Figure II-2lb; numerous voids are present also.

## MICROSTRUCTURAL STUDIES

Studies were conducted to investigate the base metal-filler metal reactions that occurred during brazing and their effect on the microstructure of the brazed joints. The approximate composition of some phase constituents was also determined.

The microstructure of a typical area of the joint between the tube and sleeve in Section MCS-2-4 is shown in the unetched condition at a magnification of 500X in Figure II-22. Considerable reaction between the base metal and the molten filler metal occurred during brazing. In Section MCS-2-4, measurements indicated that a layer of base metal (about 0.0005-inch-thick) was dissolved by the molten filler metal to form another phase at the joint interface (areas labelled A in Figure II-22). Presumably, this is a coppernickel phase because copper and nickel form a continuous series of solid solutions. The other elements contained in the base metal have a limited solubility in the molten constituents of the filler metal. This phase appears to disperse also in the area between the point interfaces (areas labelled B in Figure 11-22).

The microstructure of the brazed joint was studied by electron microprobe analysis. An area\* similar to that shown in Figure II-22 was scanned with the electron microprobe to determine the distribution of the elements in the filler metal (silver and copper) and the major elements in the 304L stainless steel base metal (nickel, chromium, and iron). The resulting X-ray images for these elements are shown in Figures II-23a through II-23e. By examining the variations in intensity that indicate the approximate concentration distribution of each element in the area being scanned, it was possible to estimate the approximate composition of phase constituents in the areas of interest. The results of these studies are discussed below.

- (1) The phase (A in Figure II-22) along the joint interface that was produced by dissolution of the base metal by the molten filler metal contained 73 percent (or more) copper, some silver (about 6 percent), and a small amount of nickel. Chromium and iron were not detected in this area.
- (2) The composition of the phases labelled B and D in Figure II-22 was almost identical to that of the phase labelled A. These particles resulted from the base metal-filler metal reaction and became detached as dissolution of the base metal progressed.
- (3) The light areas (C in Figure II-22) are composed of essentially pure silver.
- (4) The presence of nickel, chromium, and iron in the base metal (E in Figure II-22) is indicated by the X-ray images for these elements.

<sup>\*</sup> The area scanned by the electron microprobe differed slightly from that shown in Figure II-22, because the specimen was repolished before study. However, the area scanned contained the features indicated in this figure.

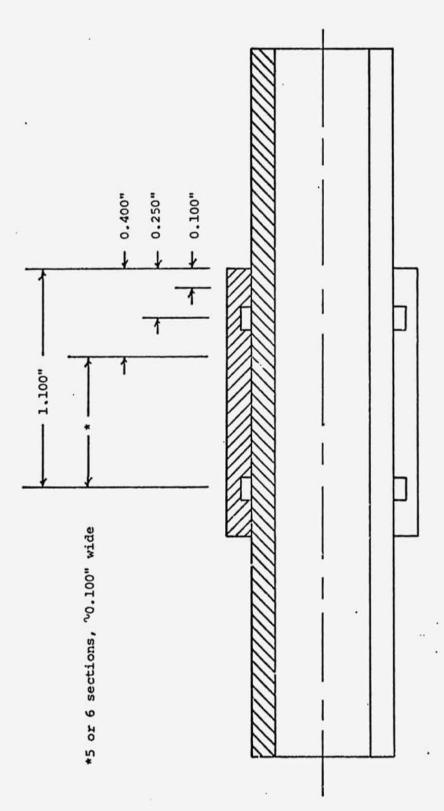


FIGURE II-1. SECTIONING PLAN FOR GROUND CHARACTERIZATION SAMPLES MCS-1, MCS-2, AND MCS-3

Sections to be cut at indicated distances from right end of sample.

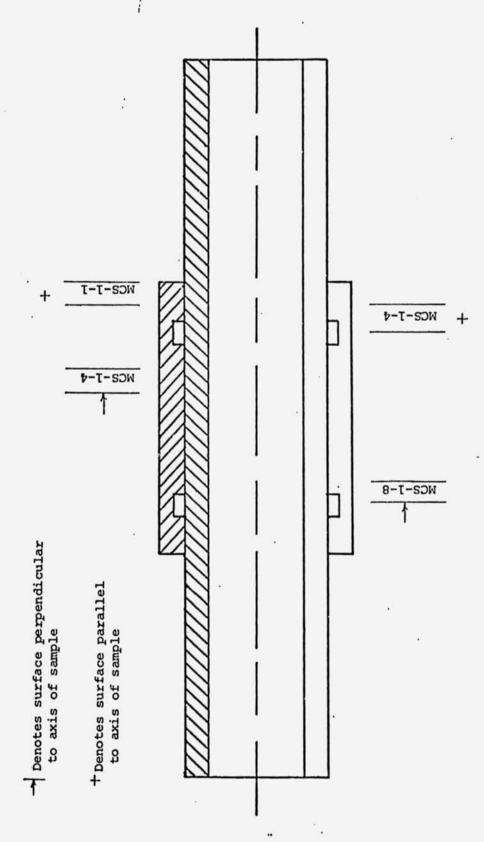
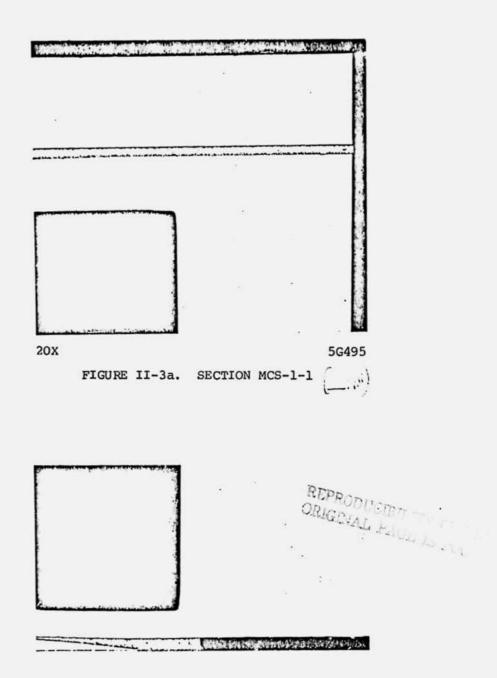


FIGURE 11-2. SURFACES EXAMINED DURING METALLOGRAPHIC EVALUATION (MCS-1)



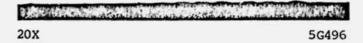


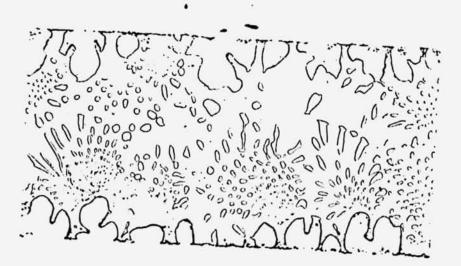
FIGURE II-3b. SECTION MCS-1-1



250x

5G483

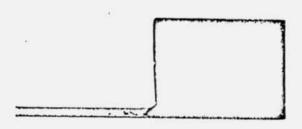
FIGURE II-4a. CECTION MCS-1-1



500X

5G484

FIGURE II-4b. SECTION MCS-1-1



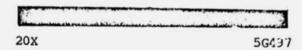
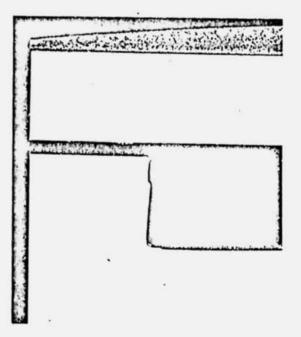


FIGURE II-5a. SECTION MCS-1-2

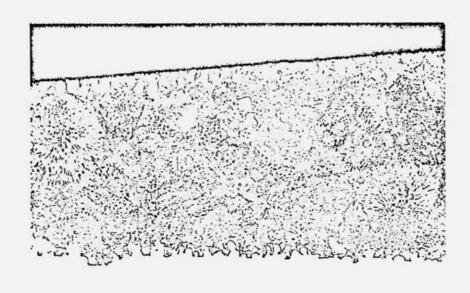


PEPRODUCIBILITY OF THE

20X

5G498

FIGURE II-5b. SECTION MCS-1-2



5G485

FIGURE II-6a. SECTION MCS-1-2

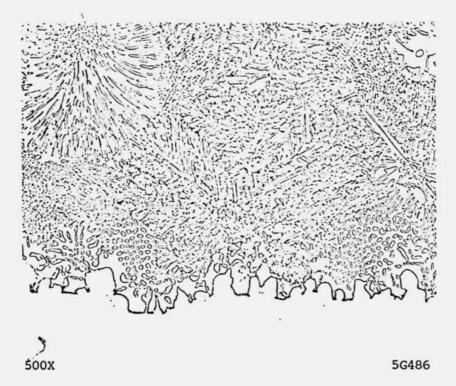


FIGURE II-6b. SECTION MCS-1-2

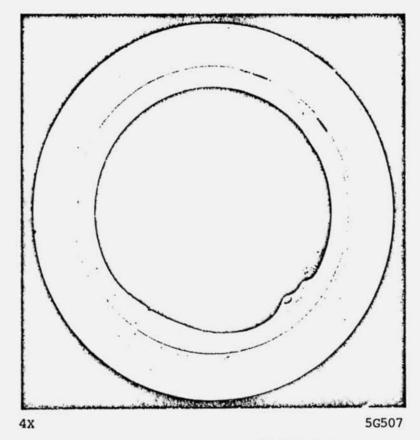
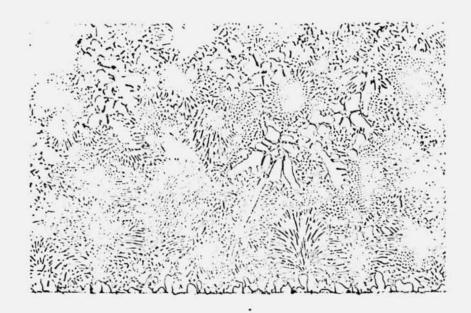


FIGURE II-7. SECTION MCS-1-4



5G489

FIGURE II-8. SECTION MCS-1-4



5G488

FIGURE II-9. SECTION MCS-1-4

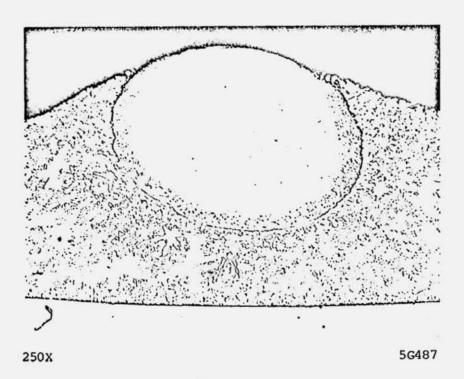


FIGURE II-10. SECTION MCS-1-4

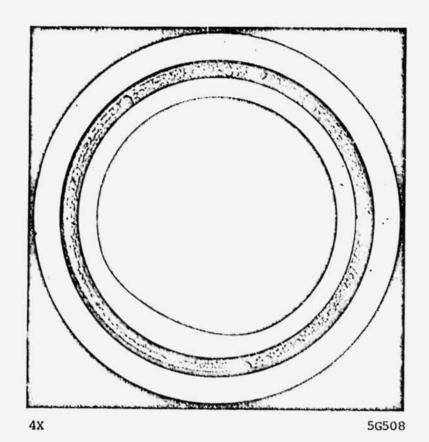


FIGURE II-11. SECTION MCS-1-9

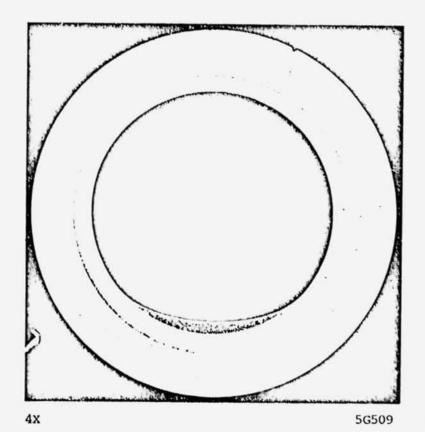


FIGURE II-13. SECTION MCS-2-4

NC2-5-4 WC2-5-1 WC2-5-9 Denotes surface perpendicular to axis of sample † Denotes surface parallel to axis of sample

FIGURE 11-12. SURFACES EXAMINED DURING METALLOGRAPHIC EVALUATION (MCS-2)



5G491

FIGURE II-14a. SECTION MCS-2-4

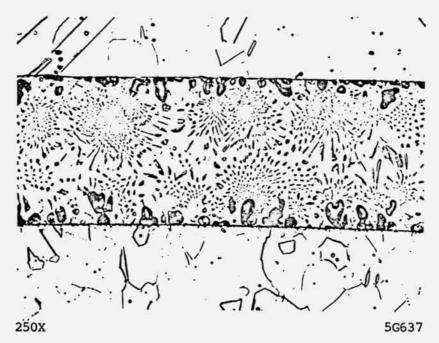


FIGURE II-14b. SECTION MCS-2-4



250X 5G490

FIGURE II-15. SECTION MCS-2-4

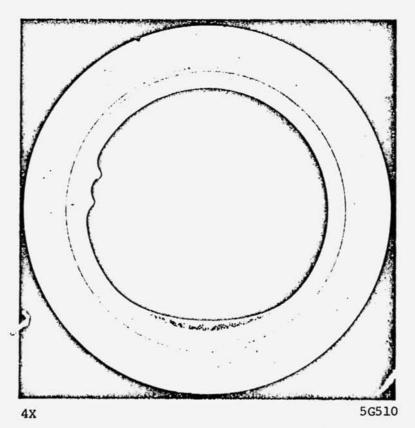
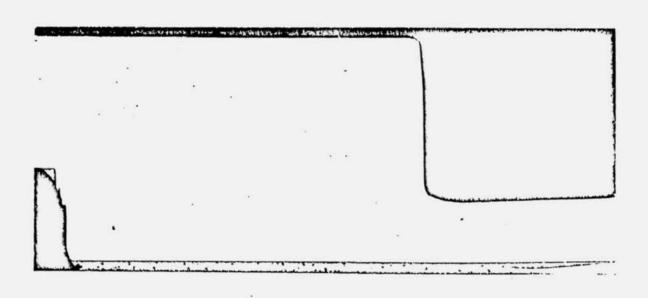


FIGURE II-16. SECTION MCS-2-7

ARICANAL PACE TO POOR



4X 5G503

FIGURE 11-17. SECTION MCS-2-9

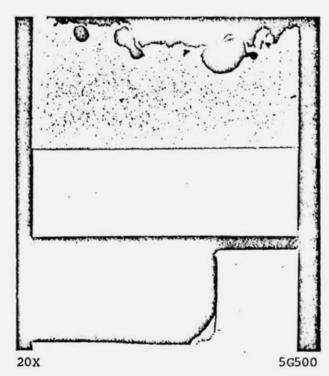


FIGURE II-18b. SECTION MCS-3-2

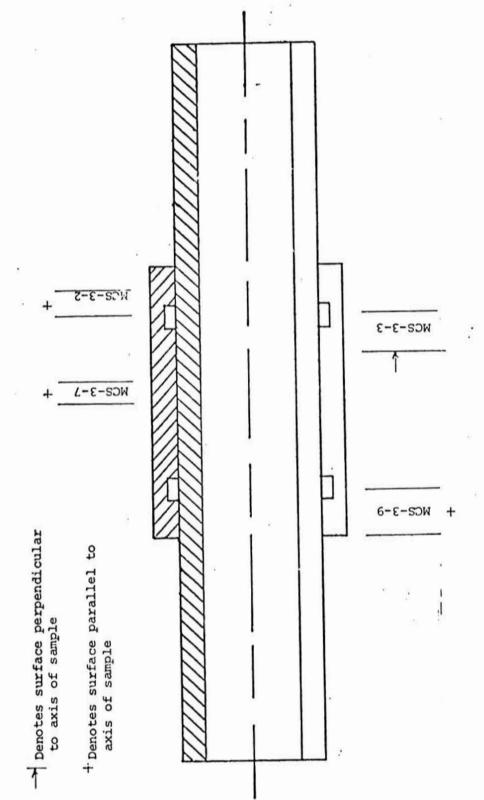


FIGURE II-18a. SURFACES EXAMINED DURING METALLOGRAPHIC EVALUATION (MCS-3)

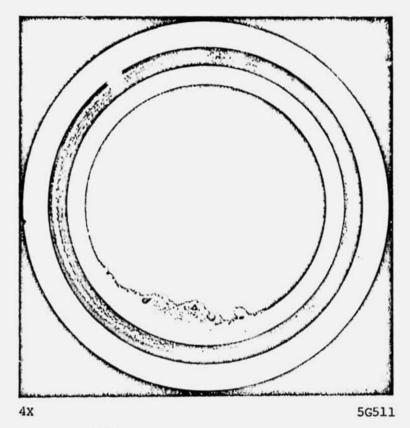


FIGURE II-19a. SECTION MCS-3-3



100X 5G494 ·

FIGURE II-19b. SECTION MCS-3-3

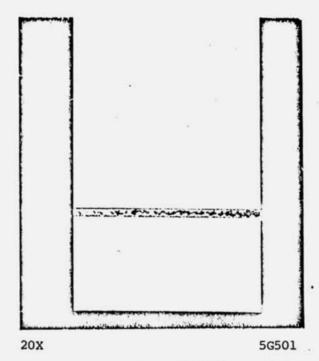


FIGURE II-20a. SECTION MCS-3-5

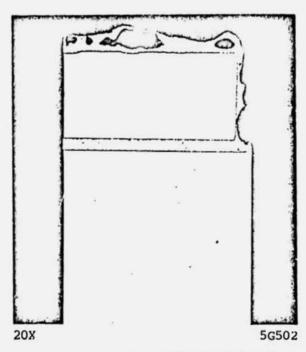
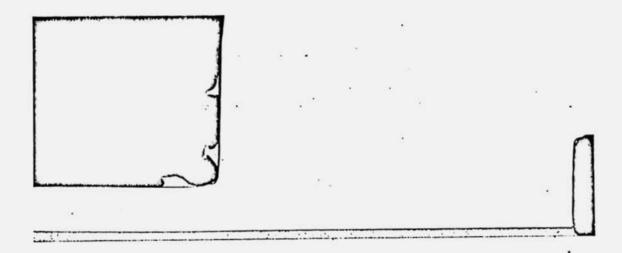


FIGURE II-20b. SECTION MCS-3-5



20X 5G506

FIGURE II-21a. SECTION MCS-3-9

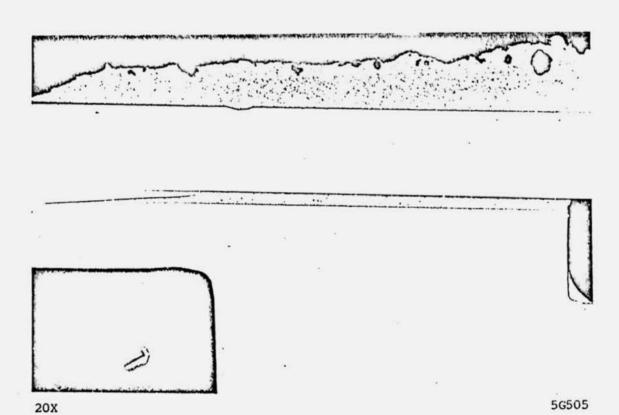


FIGURE II-21b. SECTION MCS-3-9

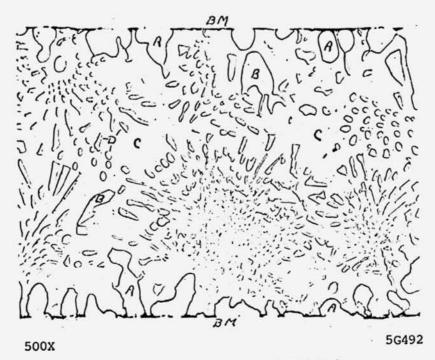


FIGURE II-22. SECTION MCS-2-4

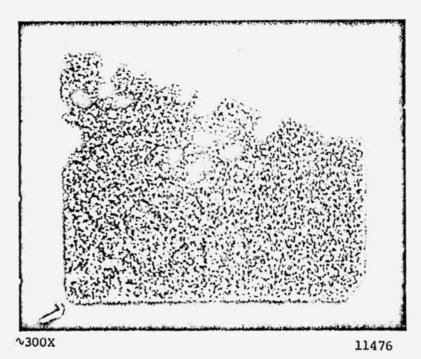


FIGURE II-23a. SECTION MCS-2-4 SILVER X-RAY IMAGE

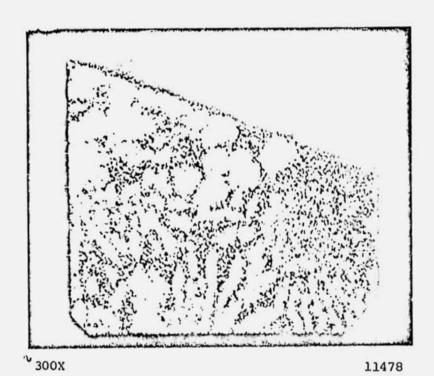


FIGURE II-23b. SECTION MCS-2-4 COPPER X-RAY IMAGE

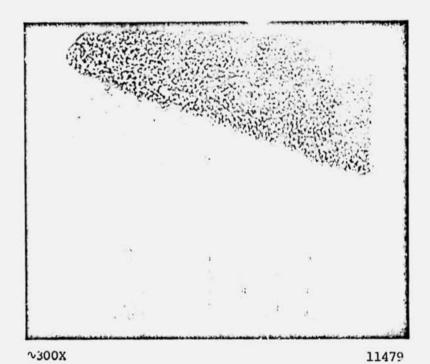


FIGURE II-23b. SECTION MCS-2-4 NICKEL X-RAY IMAGE

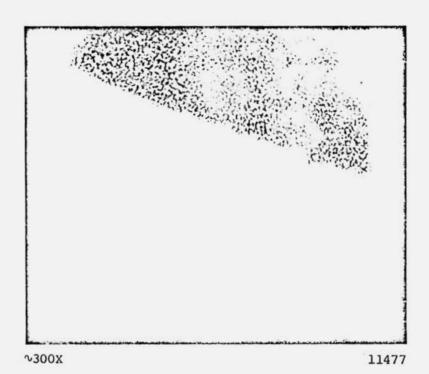


FIGURE II-23d. SECTION MCS-2-4 CHROMIUM X-RAY IMAGE

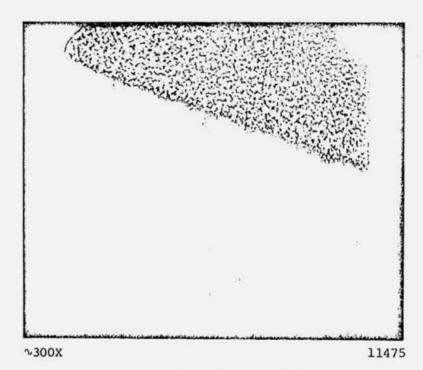


FIGURE II-23e. SECTION MCS-2-4 IRON X-RAY IMAGE